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IS 13600 (1992): Chemical analysis of silver palladium alloys [MTD 10: Precious Metals]

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भारतीय मानक

चाँदी-पेलेडियम मिश्रधातुओं के रासायनिक विश्लेषण की पद्धतियाँ

Indian Standard

CHEMICAL ANALYSIS OF SILVER PALLADIUM ALLOYS

UDC 669·225' 234 : 543 [546 : 57' 98]

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 1992

Price Group 1

Precious Metals Sectional Committee, MTD 10

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Precious Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

Silver-palladium alloys in the form of wires and strips, are used as electrical contacts in telecommunication equipment and other similar lower current applications. These alloys mainly consist of silver and palladium alongwith platinum group metals and base metal impurities. The purpose of this standard is to lay down method for chemical analysis of the main constituents i. e. silver and palladium in these alloys.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

CHEMICAL ANALYSIS OF SILVER PALLADIUM ALLOYS

1 SCOPE

This standard describes the method for determination of silver and palladium in silver-palladium alloys, in the ranges given below:

Element	*Percent by Mass*
Silver	40-70
Palladium	30-60

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
265 : 1987	Hydrochloric acid (*third revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)

3 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070 : 1992) shall be employed for the test.

4 SAMPLING FOR CHEMICAL ANALYSIS

Take 5 g of composite sample from the material by chipping from the entire cross section of the wire/strip. Remelt and pour into a mould of convenient size. This piece is then drilled or sawed from the entire cross section.

5 DETERMINATION OF SILVER BY GRAVIMETRIC METHOD

5.1 Outline of the Method

The sample is dissolved in nitric acid. Silver is precipitated as silver chloride, filtered, dried and weighed as silver chloride.

5.2 Reagents

5.2.1 *Dilute Nitric Acid*, 1 : 1 and 1 : 99 (*v/v*).

5.2.2 *Dilute Hydrochloric Acid*, 1 : 10 (*v/v*) or 1 N.

5.2.3 *Dilute Ammonium Hydroxide*, 1 : 1 (*v/v*).

5.2.4 *Agua Regia*

Mix one volume of nitric acid (rd = 1·42) with three volumes of hydrochloric acid (rd = 1·16).

5.3 Procedure

5.3.1 Transfer 0·5 g of the accurately weighed sample to a 250 ml beaker. Add 20 ml of nitric acid (1 : 1). Heat on a hot plate and boil gently till the aliquot is free from brown fumes. Cool, dilute to 50 ml and allow to stand for about 1 hour.

5.3.2 Filter through medium textured filter paper. Transfer the precipitate quantitatively on the filter paper using dilute nitric acid (1: 99). Wash the precipitate five times with 5 ml portions of dilute nitric acid (1 : 99) and then twice with water till free from acid. Transfer the filter paper and residue into a silica crucible (30 ml capacity or less) and reserve for further treatment as in **5.3.5**.

5.3.3 Adjust the volume of the filtrate to 100 ml and heat to boiling. Add dropwise 20 ml of dilute hydrochloric acid (1 : 10) with vigorous stirring. Cover the outside of the beaker with dark paper and digest on a water bath for about 2 hour. Test the supernatent liquid for completion of precipitation by adding dilute hydrochloric (1 : 10). Add more quantity of hydrochloric acid, if necessary, to complete precipitation of silver chloride.

5.3.4 Decant the solution through medium textured filter paper. Wash the precipitate twice with warm dilute nitric acid (1 : 99), decanting through the filter after each wash. Transfer the precipitate to the filter and wash with dilute nitric acid (1 : 99), till washings are free from chlorides. Reserve the filtrate for determination of palladium.

5.3.5 Dissolve the silver chloride precipitate on the filter paper **5.3.4** with ammonium hydroxide (1 : 1). Wash the filter first with dilute ammonia and then several times with hot water. Reserve the filtrate and washings of further treatment under **5.3.9**. Dry the filter paper, after transfering to the crucible containing residue reserved under **5.3.2**, char and ignite in a muffle furnace at 600°C. Cool.

5.3.6 Transfer the residue into a 250 ml beaker and treat with 100 ml aqua-regia. Heat on a hot plate. When the dissolution is complete, cool, wash down the sides of the beaker and dilute to 100 ml. Allow to stand for about 3 hours.

5.3.7 If any precipitate appears, filter through medium textured filter paper and wash several times with dilute nitric acid (1 : 99). Reserve the filtrate for the determination of palladium.

5.3.8 Dissolve the precipitate on the filter paper with dilute ammonium hydroxide (1 : 1). Wash the paper twice with dilute ammonia and then with hot water. Add the filtrate and washings already reserved under **5.3.5**.

5.3.9 Acidify the filtrate with dilute nitric acid (1 : 1) with constant and vigorous stirring, till acidic and then add 1 ml in excess. Add 1 ml of dilute hydrochloric acid (1 : 10), stir and allow to stand for 3 hours in a dark place.

5.3.10 Filter through medium textured filter paper and wash several times with dilute nitric acid (1 : 99) and preserve the filtrate for determination of palladium.

5.3.11 Dissolve the precipitate on the filter paper with dilute ammonium hydroxide (1 : 1). Wash the paper twice with dilute ammonium hydroxide (1 : 1) and then with hot water.

Acidify the filtrate with dilute nitric acid (1 : 1) with constant stirring till acidic and then add 1 ml of dilute nitric acid (1 : 1) in excess. Add 1 ml of dilute hydrochloric (1 : 10) and allow to stand for 3 hours.

5.3.12 Decant the supernatent liquid through a tared sintered crucible (G 4). Wash the precipitate twice with warm dilute nitric acid (1 : 99), decanting through the crucible after each wash. Transfer the precipitate quantitatively to the crucible and wash thrice with hot water.

5.3.13 Dry the crucible and precipitate first at 100°C for an hour and then at 130-150°C for 1 hour. Cool in a desiccator and weigh. Repeat heating and cooling till constant mass is obtained.

5.4 Calculation

$$\text{Silver, percent by mass} = \frac{M}{m} \times 75{\cdot}26$$

where

M = mass in g, of silver chloride; and

m = mass in g of sample taken.

6 DETERMINATION OF PALLADIUM BY GRAVIMETRIC METHOD

6.1 Outline of the Method

From the filtrate reserved under **5.3.4, 5.3.7** and **5.3.10** the palladium is precipitated with dimethylglyoxime, filtered, dried and weighed as palladium-dimethyl glyoximate.

6.2 Reagents

6.2.1 *Hydrochloric Acid,* rd = 1·16 (conforming to IS 265 : 1987).

6.2.2 *Dimethylgloyxime Solution*

Dissolve 1·0 g of dimethylglyoxime in 100 ml ethanol.

6.3 Procedure

6.3.1 Evaporate the combined filtrates from **5.3.4** and **5.3.7** to near dryness. Add 5 ml of hydrochloric acid and dilute to 50 ml with distilled water. Warm to dissolve the salts. Cool, transfer to a 100 ml volumetric flask and make up to volume.

6.3.2 Transfer an aliquot containing not more than 0·1 g of palladium to a clean scratchless 400 ml beaker and dilute to 200 ml. Adjust the acidity to 0·25 N with respect to hydrochloric acid. Add dimethylglyoxime solution, 2·5 ml for each 10 mg of palladium with stirring. Allow to stand at room temperature for about 1 hour.

6.3.3 Filter the palladium dimethylglyoximate through a tared sintered glass crucible (G 3). Test the filtrate by the addition of dimethylglyoxime solution to ensure completion of precipitation. Transfer the precipitate quantitatively to the crucible. Wash the orange yellow precipitate of palladium dimethylglyoximate thoroughly, first with cold water and then with hot water.

6.3.4 Dry the crucible and precipitate in air oven at 110°C for 2 hours. Cool in a desiccator and weigh. Repeat drying and weighing till constant mass is obtained. Weigh as palladium dimethylglyoximate.

6.3.5 *Calculation*

$$\text{Palladium, percent by mass} = \frac{M_1}{m_1} \times 31{\cdot}61$$

where

M_1 = mass in g, of palladium dimethylglyoximate; and

m_1 = mass in g, of the sample in the aliquote taken.

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices :		Telephone
Central :	Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	331 01 31 331 13 75
Eastern :	1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola CALCUTTA 700054	37 84 99, 37 85 61 37 86 26, 37 86 62
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Southern :	C. I. T. Campus, IV Cross Road, MADRAS 600113	235 02 16, 235 04 42 235 15 19, 235 23 15
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